

(11) (A) No. 1,117,579

(45) ISSUED 820202

(52) CLASS 309-3

(51) INT. CL. ³ A05B 3/02

(19) (CA) **CANADIAN PATENT** (12)

(54) ELECTRICALLY CONDUCTIVE CARBON FIBER-REINFORCED
CEMENT HEATING ELEMENT

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(21) APPLICATION No. 316,531

(22) FILED 781120

(30) PRIORITY DATE U.K. (49500/78) 780124

NO. OF CLAIMS 36

ABSTRACT OF THE DISCLOSURE

FEB 2 1982

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A carbon fiber-reinforced, electrically-conductive cement which can be employed in the form of floor or wall panels, or other structures, to heat a room or other area, such as aircraft runways, driveways, roads, and the like.

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BACKGROUND OF THE INVENTION

This invention relates to carbon fiber-reinforced cement heating elements. More particularly, this invention relates to carbon fiber-reinforced, electrically-conductive cement which can be employed in the form of floor or wall panels, or other structures, to heat a room or other area, such as aircraft runways, driveways, roads, and the like.

Heated floors and other structures have heretofore been prepared by laying electric cables in specific paths between terminals located in specific positions on structural support members. The heating was thus necessarily localized in that the temperature of the structure varied and was lower with increasing distance from the electric cable. Moreover, if any part of the electric cable or terminals of such structures were accidentally damaged, the entire heating unit became useless and could only be repaired with extreme difficulty and at great expense.

SUMMARY OF THE INVENTION

In accordance with the present invention, a carbon fiber-reinforced, electrically-conductive cement is provided which can be employed in the form of floors, walls and other structures to heat a room or other area.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a cross-sectional top view of a

carbon fiber-reinforced, electrically-heated cement floor panel produced in accordance with the invention.

Figure 2 is an elevated cross-sectional view of the floor panel shown in Figure 1 with an electrically insulated conductive barrier in place above it.

DESCRIPTION OF THE PREFERRED EMBODIMENTS

Because the carbon fiber-reinforced, electrically-conductive cement bodies of the present invention serve as both the structural component and heating element of the floors or other structures in which they are employed, they constitute a decided improvement over previous electrically-heated floors and structures wherein a distinct heating element and structural component were required. As a result, they are considerably cheaper to produce than such previous electrically-heated floors and structures.

Any hydraulic cement can be used in the cement compositions employed to produce the carbon fiber-reinforced, electrically-conductive heating elements of the present invention. Aggregate filler material may be employed together with the hydraulic cement in amounts conventionally employed. If a filler is employed, however, it is preferably a fine non-abrasive aggregate material, such as fly ash, and does not exceed twenty parts by weight of the total weight of cement and aggregate material.

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The fibers used in the cement compositions employed in the production of the electrically-conductive, reinforced cement heating elements of the present invention are high modulus, high strength carbon fibers. These fibers act not only to provide structural strength to the bodies in which they are employed, but also to carry the electric current through the bodies. Such fibers are commercially available and can be prepared as described in U.S. Patents 3,454,362, 3,412,062 and 4,005,183. The term "carbon" 10 as used herein is intended to include graphitic and non-graphitic fibers.

The length of the carbon fibers employed may be varied to suit requirements, typical lengths being 5 mm. to 75 mm. The thickness of the fibers may vary from about 5 microns to about 25 microns, but is usually within the range of about 7 microns to about 9 microns.

The amount of fibers employed is such as to obtain the desired characteristics, typically from 1 part by weight to 6 parts by weight of fibers per 100 parts 20 by weight of the "dry components" of the cementitious composition. By "dry components" in this context is meant the cement and other solid aggregate filler material (if present) which together make up the cementitious composition, but not including the carbon fiber itself. Most usually the fibers are present in an amount of from 2.5 parts by weight to 5 parts by weight per 100 parts by weight of the dry materials.

The water, of course, must be employed in an amount sufficient to hydrate the cement. In order to produce a cementitious product having maximum strength, however, the amount of water should be held to a minimum consistent with this purpose. Typically, from about 25 parts by weight to about 55 parts by weight, preferably from about 30 parts by weight to 45 parts by weight, of water per 100 parts by weight of the dry components in the mix are employed.

10 Because staple fibers are employed, a small amount of polyethylene oxide may be added to the mixture in order to facilitate incorporating the fibers therein. Preferably, such polyethylene oxide is added in an amount sufficient to not only prevent clumping of the fiber strands or bundles employed, but also to effect separation of the individual fibers of such strands or bundles and more or less uniformly dispose them throughout the mix, i.e., to deflocculate the fiber strands or bundles. Generally, the polyethylene oxide is employed in an amount equal to at least 0.14 part by weight per 100 parts by weight of the dry components employed. Excessive amounts of polyethylene oxide do not appear to have any beneficial effects. For this reason, amounts of polyethylene oxide in excess of about 0.50 part by weight per 100 parts by weight of the dry components present are unnecessary.

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The polyethylene oxide suitable for use in the invention is water soluble, has a molecular weight from about 500,000 up to about 5,000,000, and is commercially available from Union Carbide Corporation under the trademark "Polyox." Grade WSR-301 has been found to be especially suitable. This grade has a molecular weight of about 4,000,000.

Mixing of the components of the cementitious composition, including the cement itself, aggregate material (if present), reinforcing carbon fibers, polyethylene oxide additive (if present), and the required amounts of water, can be effected using conventional techniques. To facilitate dissolution of the polyethylene oxide, it is preferred to add this material to the water before it is admixed with the other components of the mixture. The carbon fibers are preferably added last, in a gradual manner and with stirring to assist in obtaining a smooth mix.

After formulation of the cementitious composition, as described above, it is cast or moulded into slabs, or other bodies, of any convenient size and thickness suitable for use, for example, as a floor or wall panel, or other heating element, and allowed to set under suitable conditions. For ease of formation and handling, such slabs are typically 2-3 feet in length and width, and 0.5-1.5 inches thick. Electrical terminals are incorporated into the

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composition, usually before it is fully cured, as described below, to provide means for supplying an electrical current to the slab after it has fully cured. In order to provide sufficient resistance so that the slab can be operated on a standard 115-120 volt power supply, and to ensure that the slab is heated over its entire surface, a series of electrically insulating strips are set into the cement in a manner which forces the current to traverse a longer path between the terminals. By changing the cross-sectional area of the slab and/or the length of the electrical circuit through it, it is possible to adjust the resistance of the slab to meet various design requirements.

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The electrically insulating strips which are incorporated into the cement to increase the path of the current through the slab are preferably extended into the slab from two opposite sides in a manner which causes the current to flow back and forth from one side of the slab to the other as it traverses its way across the slab from one terminal to the other. Such current flow can be achieved by positioning insulating strips between the terminals and alternating them so that they extend into the slab first from one side to beyond the middle of the slab, and then from the opposite side to beyond the middle of the slab. For maximum flow, the strips should extend at least three-quarters of the way across the slab, and most preferably a full 90 per cent of the way across the entire length of the slab. In any event, the current is

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preferably forced to flow at least five times, preferably at least 8 times, the distance it would flow in the absence of the strips. These strips can be made of any convenient insulating material and may be incorporated into the slab in any convenient manner, e.g., by tacking them to the bottom of the form in which the slab is cast.

Electrical terminals must also be incorporated into the slab to provide means for transmitting current from the power supply to the carbon fibers in the slab.

10 However, if insufficient contact is provided between the terminals and the randomly distributed fibers of the slab, narrow current paths are produced which results in overheating and arcing. For this reason, it is necessary to provide terminals which have contact with a sufficient number of fibers to transmit the current from the power source to the fibers without creating "hot spots". This problem has been resolved by embedding reservoirs filled with a conductive resin system at opposite ends of the slab and introducing conductive wire leads into contact with the resin. Such reservoirs may be of any suitable size and shape, typically one inch to two inches in length and one-eighth inch to one-quarter inch in width. The resin system employed should be such as will both conduct an electrical current and effectively bond the conductive leads and the cement particles.

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In order to render the resin system employed electrically conductive, it is usually necessary to add

an electrically conductive filler thereto, e.g., chopped carbon fibers, carbon black, and the like. Generally, from 5 parts by weight to 50 parts by weight, preferably from 10 parts by weight to 20 parts by weight, of such electrically conductive filler per 100 parts by weight of the resin system are employed.

For convenience of operation, the resin system employed should be hydrophobic, liquid, insoluble in water and capable of curing at room temperature simultaneously with the cement. While many resins are not hydrophobic, they may, nevertheless, be employed provided they become hydrophobic when a hardening agent is added thereto. The hydrophobic character of such resin system causes the expulsion of water present on the surfaces of the discrete cement particles in the vicinity of the resin which might otherwise interfere with the curing and bonding of the resin to these particles. As a result, an improved bond is effected between these particles and the conductive wires in contact with the resin system.

20 An epoxy resin system is preferably employed to bond the conductive wires to the cementitious composition because such system is easy to handle and capable of being cured at room temperature. Such system comprises an epoxy resin together with a reactive epoxy resin hardener in an amount conventionally used in the art to cure epoxy resins.

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The epoxy resins which are preferably employed to bond the conductive wires to the cementitious composition are the liquid polyglycidyl ethers of polyhydric phenols, particularly the liquid diglycidyl ethers of bis(4-hydroxyphenyl)methane and bis(4-hydroxyphenyl)-dimethylmethane. Such resins are usually produced by the reaction of epichlorohydrin with a polyhydric phenol in the presence of a base.

As is well known, by varying the proportions 10 of reactants employed in producing an epoxy resin it is possible to produce a product varying in viscosity, molecular weight, and hydroxyl content. The resins employed in the invention are those low molecular weight, low viscosity, liquid epoxies in which, most preferably, the main or predominant constituent is free of hydroxyl groups, e.g., those epoxies in which the reaction product of two moles of epichlorohydrin with one mole of dihydric phenol is the main or predominant constituent. While hydroxyl groups are usually present in most all commercially available 20 epoxy resins, many are available which have a low hydroxyl content, and resins of this type are most preferred for use in this invention. Especially preferred resins of this type are the diglycidyl ethers of bis(4-hydroxyphenyl)methane and the diglycidyl ethers of bis(4-hydroxyphenyl)dimethylmethane.

The hardening agent employed together with the epoxy resin must be one which forms a hydrophobic system with the resin and causes it to cure at room temperature. The room temperature cure is necessary to allow the resin to cure simultaneously with the cement, and the formation of a hydrophobic system is necessary to displace water present on the surfaces of the discrete cement particles in the vicinity of the resin which might interfere with the curing and bonding of the resin to these particles.

10 In this manner, an improved bond is effected between these particles and the conductive wires embedded in the resin system.

Among the epoxy resin hardeners which can be employed in the present invention are those hardeners sold as "Ancamine"** R (manufactured by Anchor Chemicals Co. U.K. Ltd.) and "Sur-Wet"** R (manufactured by Pacific Anchor Chemicals Co.). Both of these hardeners are hydrophobic fatty amines having an amine number of from 170 to 180.

When admixed with an epoxy resin, the hydrophobic character of these hardeners is imparted to the entire resin system.

For safety purposes an electrically insulated conductive barrier should be provided above the conductive portion of the slab. Such barrier functions as a safety

*"Ancamine" is a registered trademark of Anchor Chemicals Co. U.K. Ltd.

**"Sur-Wet" is a registered trademark of Pacific Anchor Chemicals Co.

system to prevent electric shock in the event that slab is inadvertently penetrated by a conductor such as workman's tools, nails, etc. Conveniently, the barrier may be in the form of an electrically grounded metal foil embedded between two thin layers of cement, e.g., from one-eighth inch to one-quarter inch in height. The metal foil may typically be aluminum, or other conductor, provided with an electric ground. Alternatively, or indeed in addition to the metal foil, a doubly insulated transformer may be used. The cement may be formed from the same constituents as the conductive portion of the slab except that the carbon fibers are eliminated so as to render the barrier non-conductive.

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Referring now to the drawings, an electrically-heated floor panel suitable for heating a room or other area is shown in Figures 1 and 2. The floor panel 10 is 25" x 25" x 1" and composed of carbon fiber-reinforced cement 11. Embedded in the cement are cylindically-shaped reservoirs 12 and 13. The reservoirs are 2 inches long and one-quarter inch in diameter, and are filled with an electrically-conductive epoxy resin system which acts as terminals for conductive copper wire leads 14 and 15 which are encapsulated therein. Wire leads 14 and 15 are connected to a power supply, not shown. The epoxy resin system in reservoirs 12 and 13 is rendered electrically conductive by the addition of carbon fibers and carbon black. Insulating strips 16 are positioned between the terminals and extend into the slab from

opposite sides thereof in a manner which causes current introduced into the slab to flow back and forth from one side of the slab to the other as it traverses its way from one terminal to the other. The insulating strips are composed of a melamine laminate 22.5 inches long and one-sixteenth inch thick, and are spaced 2.5 inches apart from each other and the ends of the slab.

To prevent electrical shock in the event the slab is inadvertently penetrated by a conductor such as workman's tools, nails, etc., electrically insulated conductive barrier 17 is provided above the conductive portion of slab 10. Insulating barrier 17 is composed of aluminum foil 18 which is embedded between cement layers 19 and 20 and connected to ground 21.

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EXAMPLE 1

A carbon fiber-reinforced cement slab was prepared by casting a formable cementitious composition produced by admixing the constituents shown in Table 1 below in a 20-gallon power operated mixer using a dough hook attachment.

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Table 1

<u>Constituent</u>	<u>Parts by Weight</u>
Cement	100.0
Carbon Fibers	3.5
Water	42.5

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The cement and water were thoroughly mixed for 3 minutes to form a smooth slurry. Handfuls of carbon fibers were then added and stirring was continued for an additional 8 minutes. The cement employed was a Portland cement conforming to British Standard 12. The fibers employed were "Thornel" VMA fiber mat. "Thornel" VMA mat is manufactured by Union Carbide Corporation and consists of carbon filaments having a length of 25 mm. to 75 mm., an average diameter of 9 microns, an average 10 Young's modulus of 35×10^6 psi. (240 GPa) and an average tensile strength of 200×10^3 psi. (1.4 GPa).

The mixed constituents were travelled into a 25" x 25" x 1" wooden mould. Melamine laminate strips had been previously tacked edgewise to the bottom of the mould so that they extended into the cementitious composition in an alternating manner from opposite sides of the mould. The strips were 22.5 inches long and one-sixteenth inch wide, and were spaced 2.5 inches from each other in the manner shown in Figures 1 and 2.

20 After the cement was cast into the mould, cylindrical grooves 2" long and 0.25 inch in diameter were hallowed out of the wet cement at opposite ends of the slabs as shown in Figures 1 and 2. These grooves were then filled with a liquid hydrophobic epoxy resin system

**"Thornel" is a registered trademark.

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containing 20 parts by weight of carbon black and 10 parts by weight of "Thornel" VMA fiber mat to render it electrically conductive. Braided copper wire leads, connected at their opposite ends to an electrical power source, were then embedded into the epoxy resin system. The epoxy resin system employed was composed of one hundred (100) parts by weight of a commercially available liquid epoxy resin produced by the reaction of epichlorohydrin and 2,2-bis(4-hydroxyphenyl)propane (Epikote 828, manufactured 10 by Shell Chemicals U.K. Ltd.) and one hundred (100) parts by weight of "Ancamine" R, a hydrophobic fatty amine epoxy resin hardening agent having an amine number of from 170 to 180. The system was both insoluble in water and capable of curing at room temperature simultaneously with the cement.

A layer of cement formed from the same constituents as the conductive portion of the slab, except that the carbon fibers were eliminated, was then applied to the top surface of the wet slab by means of a trowel to a 20 thickness of approximately one-eighth inch. A thin paper-backed aluminum foil was then bonded to the cement layer by means of the same epoxy resin system employed to bond the copper wire leads to the slab except that the carbon black and carbon fibers were eliminated. The foil, which

had been previously treated with an epoxy resin (e.g. "Epilux"⁴) to prevent caustic attack, was bonded with the aluminum uppermost. The same epoxy resin system employed to bond the foil to the cement layer was then applied to the aluminum portion of the foil, and a second cement layer was then applied to the aluminum foil to a thickness of one-quarter inch. The second cement layer was of the same composition and was applied in the same manner as the first layer.

10 The slab prepared in this manner was then allowed to cure for two days at room temperature. At the end of this time, the slab was connected to a variable transformer, the aluminum foil being electrically grounded. Satisfactory heat was produced from the slab at 117 volts. The voltage was then increased to 240 and a rheostat was employed to maintain the surface temperature of the panel at 35°C. This temperature was maintained continuously for four days, after which the panel was disconnected. The specific resistance of the panel was 1 ohm cm. and the volume resistance was 1-2 ohm/cm.³

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⁴"Epilux" is a registered trademark of Celanese Corp.

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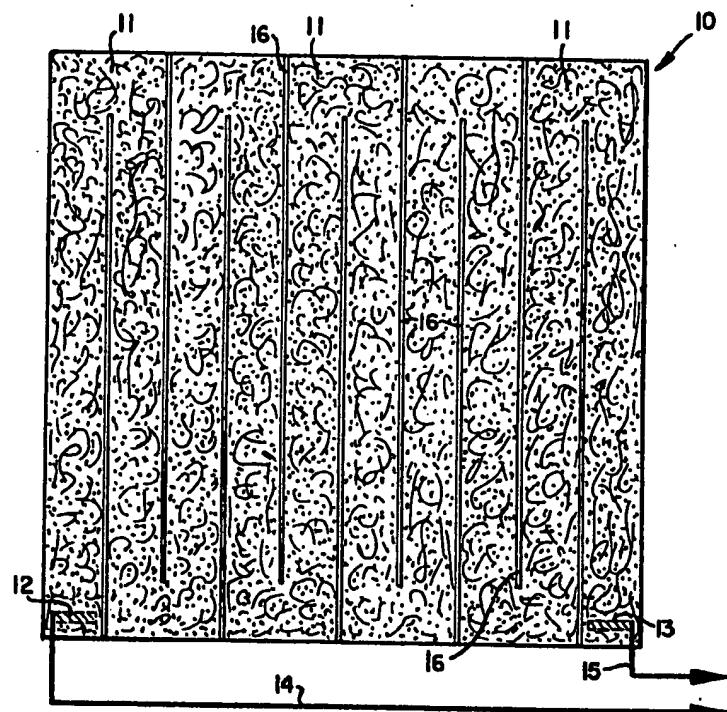


FIG. 1

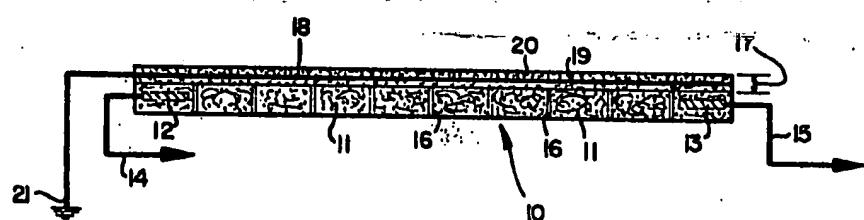


FIG. 2

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